

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 10/743,562
First Named Inventor : Renuga Gopal
TC/A.U. : 1732
Examiner : Daniels, Mathew J
Title : FIBER REINFORCED COMPOSITE AND
METHOD OF FORMING THE SAME
Filed : 12/22/2003
Confirmation No. : 5054
Docket No. : NAA 0020 PA/41049.22

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

DECLARATION PURSUANT TO 37 CFR 1.131

We,

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760154, Singapore,

KAZUTOSHI FUJIHARA, of #202, 1487-2 Sakado-ichiba, Sodegaura,
Chiba 299-0262 Japan,

POEY LING LOH, of 61, Mount Sinai Drive, #14-01, Singapore 277113,
Singapore,

WENG CHIONG KELVIN FOONG, of 25 Holland Hill, #02-05, Singapore
278740, Singapore,

VIJAY KUMAR GANESH, of Blk 210, Jurong East Street 21, #02-39, 1
Singapore 600210, Singapore,

SEERAM RAMAKRISHNA, of 49, Jurong East Ave 1, #01-02, Singapore
609781, Singapore,

CHONG LIN CHEW, of 65 Sunset Way #03-13, Freesia Woods,
Singapore 597090, Singapore,

declare as follows under penalty or perjury.

1. We are the named inventors for the above noted application (hereinafter "The Application") and as such have knowledge of the facts contained herein.
2. Gopal was a student at the National University of Singapore during the period from July 2000 to April, 2002, enrolled first in a Bachelor of Engineering program prior to June 2001 and then in a Master's program after May 2001.
3. As part of the B. Eng program, Gopal conducted research from July 2000 to May 2001. Her research project during this period was to make composite wires using rigid dies. By May 2001, Gopal realized that to make better composite wires with a small diameter, it would be desirable to reduce the stress exerted on the fibers during the formation process. Her solution was to use a flexible shrinkable die.
4. As part of her Master's program, Gopal, Kazutoshi and Ganesh researched a suitable flexible shrinkable die and a suitable procedure for forming composite wires with improved properties. Gopal and Kazutoshi obtained some flexible shrinkable tubing under the brand name SUMITUBE, and used these tubing to form fiber reinforced composite wires in a process as described and claimed in the Application, prior to October 19, 2001.
5. During this period, Kazutoshi conducted preliminary experiments to determine which types of shrinkable die would potentially provide the desired performance. Ramakrishna and Ganesh, who had expertise in the field of developing composites for biological applications, had weekly discussions with Gopal on her research progress and contributed their expertise on how to design and form a desired composite. They provided direction for Gopal's research activities. Chew, Foong and Loh, who had expertise in the field of dentistry, contributed their expertise in forming a composite wire that is suitable for orthodontic use, including the desired characteristics of an orthodontic wire. They identified the mechanical characteristics that would be desirable for a composite wire to be clinically effective. They assessed all the wires we tested from a clinical perspective and provided direction for improvement in that regard.
6. Our work during this period was memorized in a routine progress report (in the form of PowerPoint slides, hereinafter "the Report"), a copy of which is attached as Exhibit A to this Declaration. The Report was prepared by

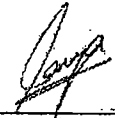
Gopal, and was shared with Ramakrishna on October 19, 2001, as part of a routine progress review of this research project.

7. The Report shows the processing setup used and the steps took for forming the composite wires (see slides 4, 9, and 11). In particular, on slides 4, 9 and 11, it is shown that the process performed included the steps of inserting glass fibers and resin into a heat-shrinkable tube, which was hung from a support bar; applying heat to shrink the tube; and curing the specimen. During the formation process, it is observed that the tunnel in the shrinkable tubing retained its cross-sectional shape and shrunk uniformly in cross-sectional area when the tubing was heat-shrunk. The cross-sectional shape of the tunnel in the tubing was round both before and after shrinkage. This is evidenced from the cross-sectional images of the composite wires produced from this process, shown on slides 5 to 7 and 10 of the Report, where the round peripheral edges of the wires reflect the round shape of the shrunk tubing tunnel. The setup and steps shown in the Report reflect embodiments disclosed and claimed in the Application (see e.g. FIGS. 1 to 4 of the Application and accompanying description). The Report is thus evidence that the invention of the Application was actually reduced to practice prior to April 18, 2002.
8. The images on slides 5 to 7 and 10 also show that the fibers were substantially evenly distributed in the composite wires produced by the process. The graphs of measured results on slides 12, 13 and 16 of the Report show that the product composite wires exhibited improved mechanical properties. These images and graphs show that the process we performed worked for the intended purposes prior to April 18, 2002.
9. Our activities described above occurred in Singapore, which is a member state of the World Trade Organization (WTO).
10. We hereby declare that all statements made herein are of our own knowledge, are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine and imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issued thereon.

Signed at

, Singapore

On this 7 day of March, 2008



RENUGA GOPAL

Signed at

On this _____ day of _____, 2008, Japan

KAZUTOSHI FUJIHARA

Signed at

On this 17 day of Mar, 2008, Singapore

POEY LING LOH

Signed at

On this 7th day of March, 2008, Singapore

WENG CHIONG KELVIN FOONG

Signed at

On this day of , Singapore
2008

VIJAY KUMAR GANESH

Signed at

On this 7TH day of March, 2008, Singapore

SEERAM RAMAKRISHNA

Signed at

On this 7th day of March, 2008, Singapore

CHONG LIN CHEW

Signed at Sodegaura-City, Chiba,
Japan
On this 16th day of March, 2008


KAZUTOSHI FUJIHARA

Signed at _____
On this _____ day of _____, Singapore
_____, 2008

POEY LING LOH

Signed at _____
On this _____ day of _____, Singapore
_____, 2008

WENG CHIONG KELVIN FOONG

Signed at _____
On this _____ day of _____, Singapore
_____, 2008

VIJAY KUMAR GANESH

Signed at _____
On this _____ day of _____, Singapore
_____, 2008

SEERAM RAMAKRISHNA

Signed at _____
On this _____ day of _____, Singapore
_____, 2008

CHONG LIN CHEW

Signed at

On this day of , Japan
2008

KAZUTOSHI FUJIHARA

Signed at

On this day of , Singapore
2008

POEY LING LOH

Signed at

On this day of , Singapore
2008

WENG CHIONG KELVIN FOONG

Signed at

On this 15 day of March, Singapore
2008

VIJAY KUMAR GANESH

Signed at

On this day of , Singapore
2008

SEERAM RAMAKRISHNA

Signed at

On this day of , Singapore
2008

CHONG LIN CHEW

EXHIBIT A



**COMPOSITE
ORTHODONTIC ARCHWIRE**

PRESENTED BY: RENUGA GOPAL

DATE: 19TH OCT 2001

CONTENTS

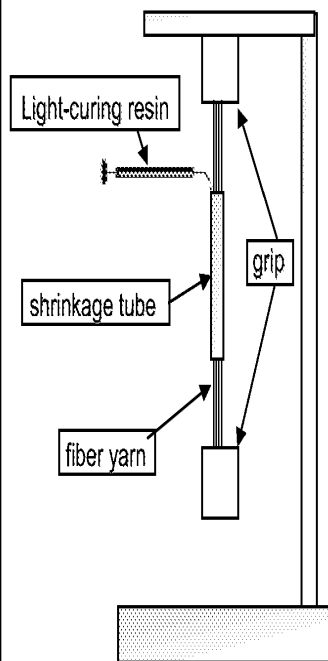
- **Material System**
 - Dental resin + GF
 - Epoxy resin + GF
- **Fabrication Method**
 - Optimal procedure
 - Surface Treatment of Glass Fibers
- **Testing**
 - Bending characteristics
 - Recovery of Wires
- **Future Work**



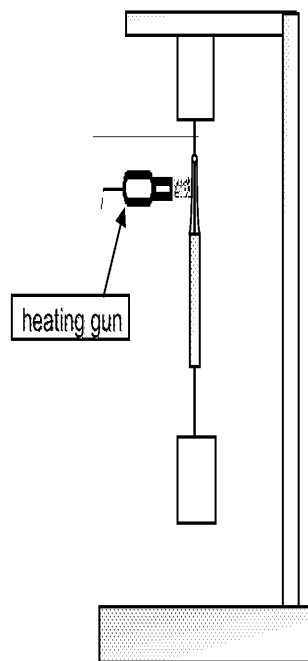
**DENTAL RESIN / GF
COMPOSITE**

Std Fabrication Method for GF/Dental Resin Wires

Glass fibers, brushed with resin, are inserted into tube and hung from a support bar. Light-curing resin injected into the tube to fill it up.

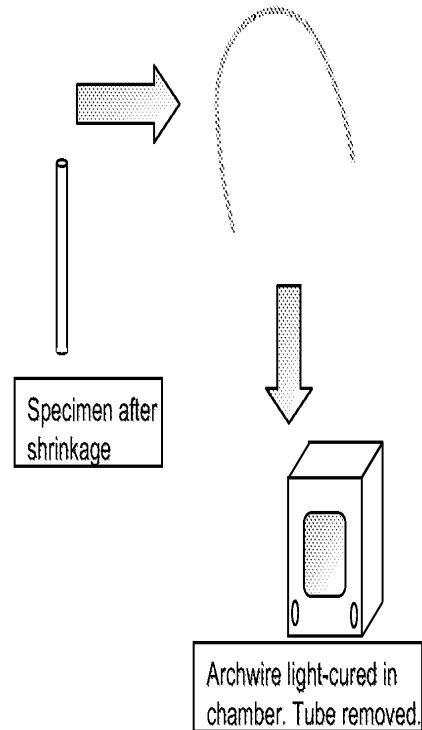


Heat applied using a heating gun. Both ends are heated. Tube vacuumed. Entire tube heat-shrunk. Excess resin is extruded out. Specimen is held in tension.

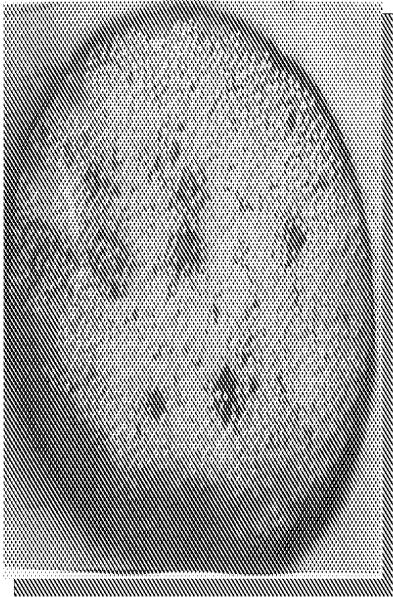


Specimen shaped into desired profile and placed in a light-curing chamber for 2 minutes. Shrinkage tube removed.

Specimen preformed into desired arch

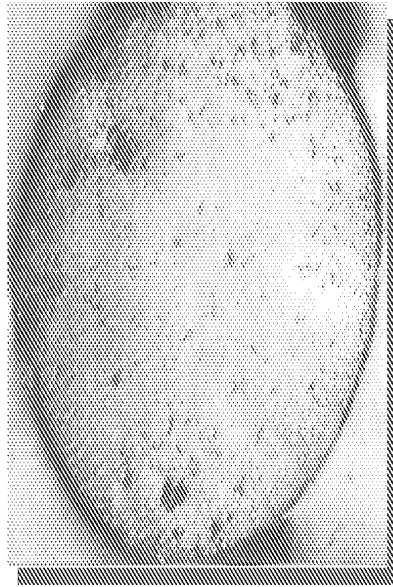


CROSS-SECTION



MTD I

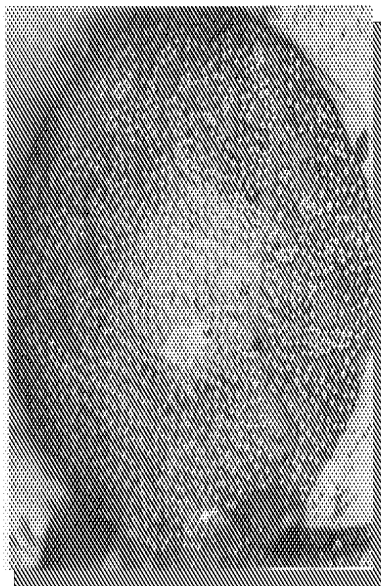
1. Insert tube and fill
2. Heat shrink



MTD II

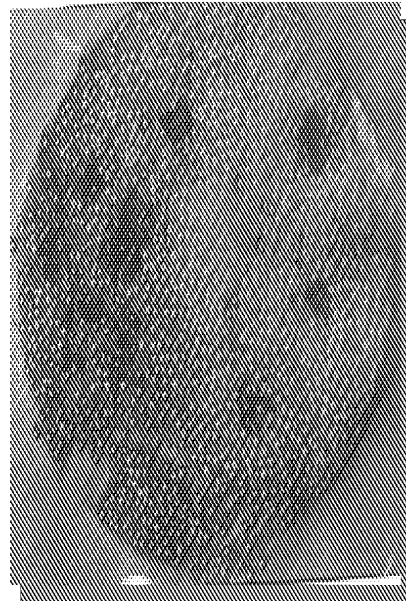
1. Brush GF
2. Insert tube and fill
3. Heat shrink

CROSS-SECTION



MTD III

1. Brush GF
2. Insert tube and fill
3. Vacuum
4. Heat shrink



MTD IV


1. Brush GF
2. Insert tube and fill
3. Heat shrink
4. Vacuum

CROSS-SECTION



MTD V

1. Brush GF
2. Insert tube and fill
3. Heat shrink the ends 1st
4. Vacuum
5. Heat shrink the whole tube

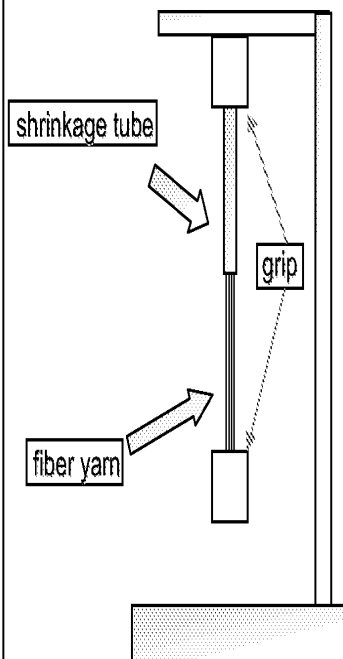


**EPOXY / GF
COMPOSITE**

Std Fabrication Method for GF/Epoxy Wires

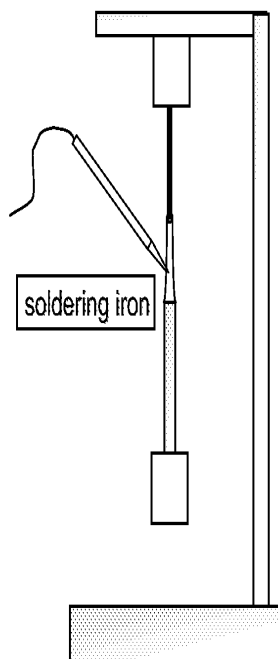
1

Hang the glass fiber from the support bar, and coat the glass fibers with the resin using a brush.



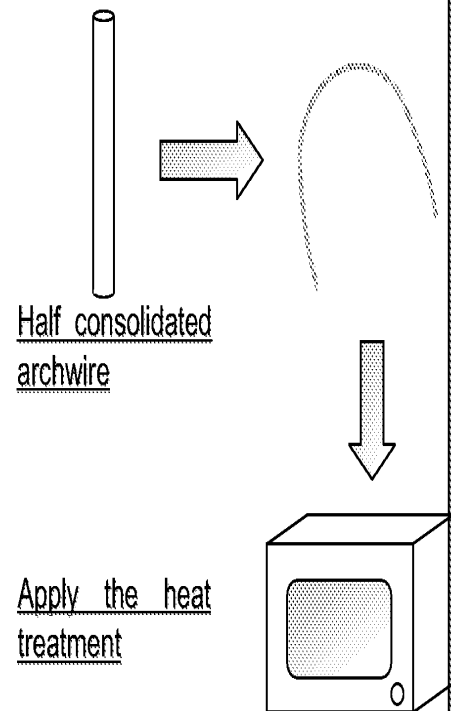
2

Apply heat using a soldering iron and extrude excess resin and voids.

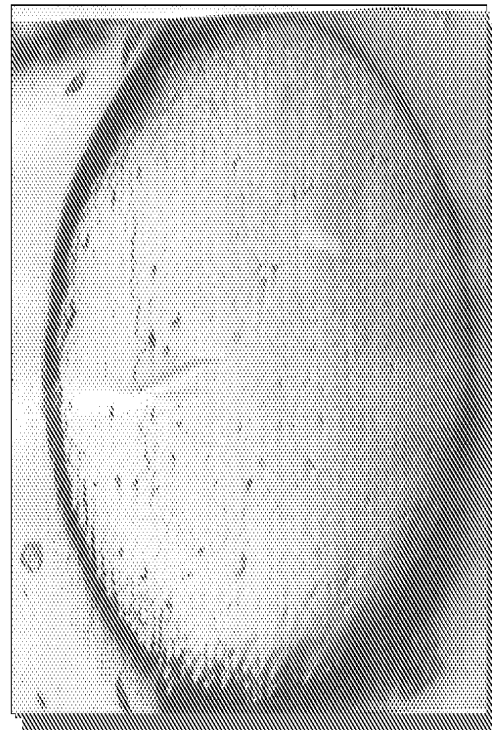
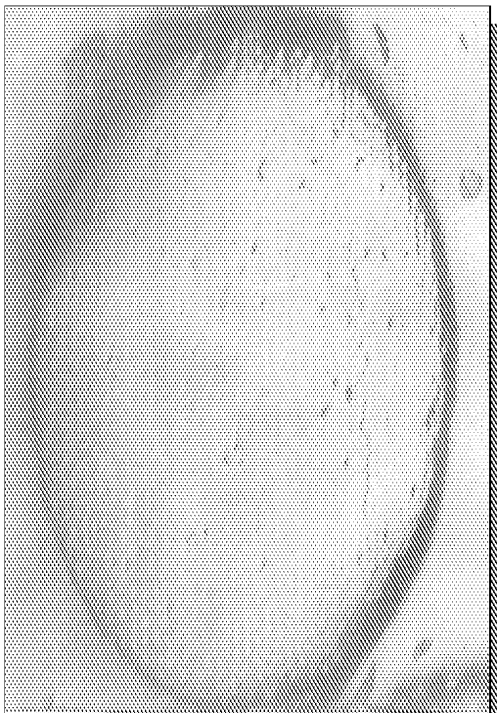


3

After 1 hour, archwire is fixed in the geometry of commercialized archwire, and then heat treatment is applied for 1 hour at 100 °C.



CROSS-SECTION

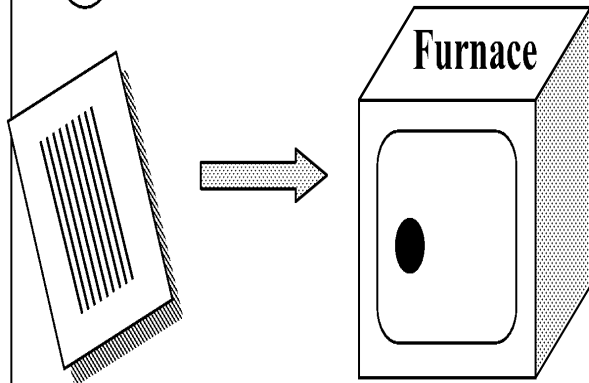


INSERT PICTURE OF SPECIMEN

EPOXY RESIN

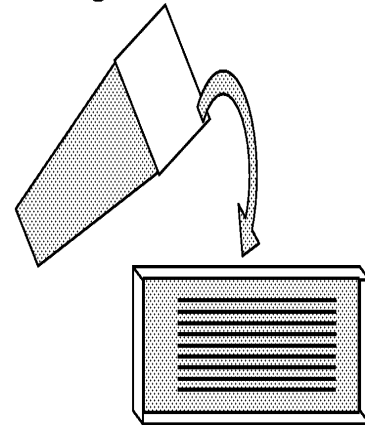
SURFACE TREATMENT

① Remove Binder



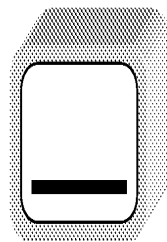
@ 400 °C; 40 hrs

② Epoxy Silane Coupling Agent Treatment



Immerse GF; 10mins

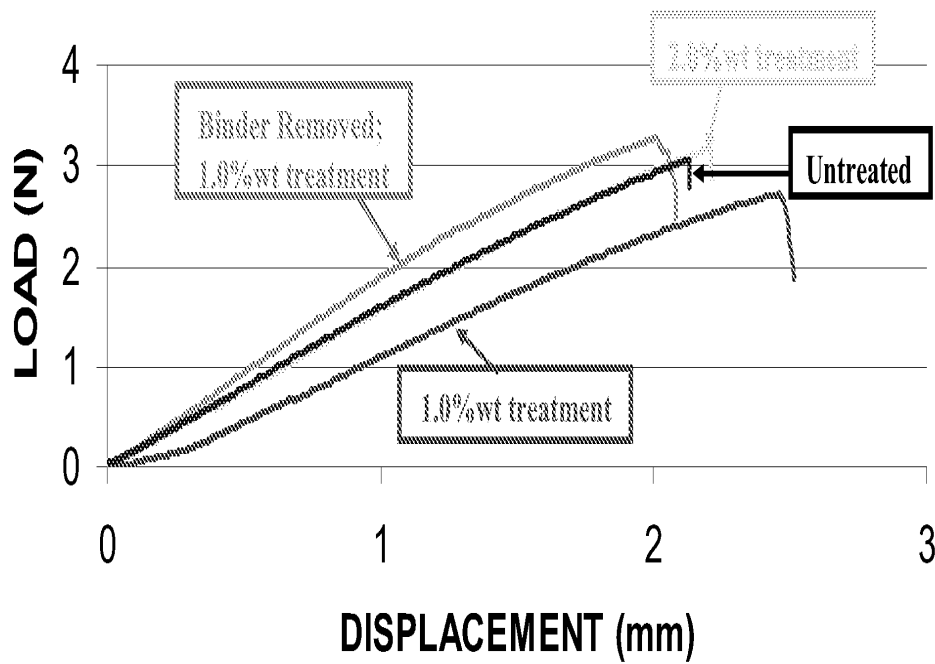
③ Oven - Dry



@ 100 °C; 20 min

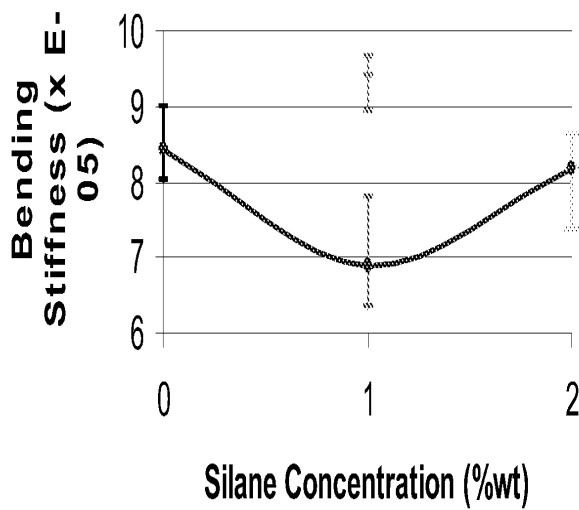
TREATMENT EFFECTS

Bending Characteristics

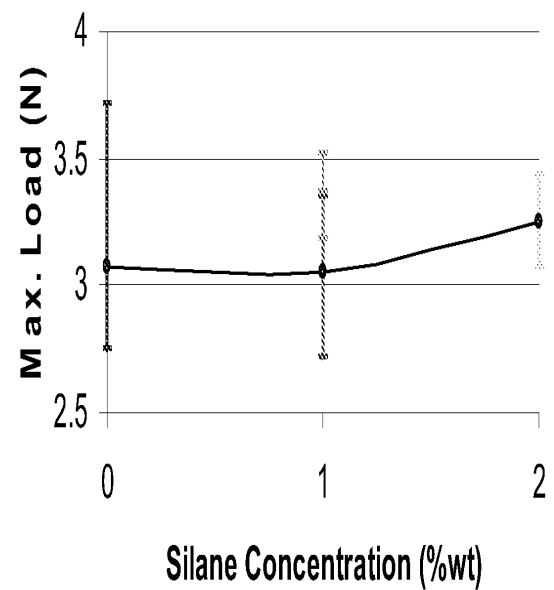


TREATMENT EFFECTS

Fiber Surface Treatment Effect



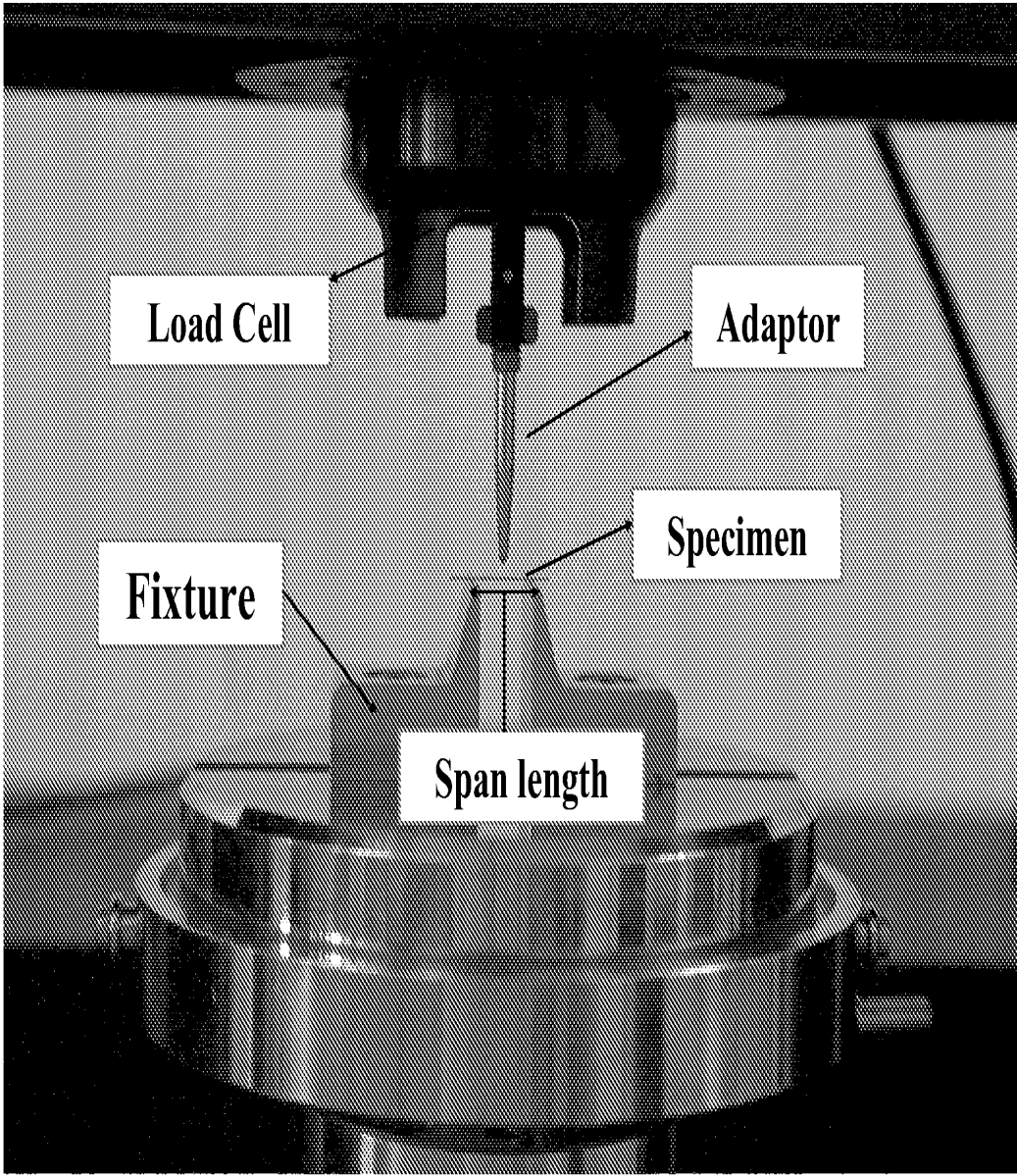
Fiber Surface Treatment Effect



- Untreated
- 1.0%wt treatment
- 2.0%wt treatment
- Binder Removed; 1.0%wt treatment

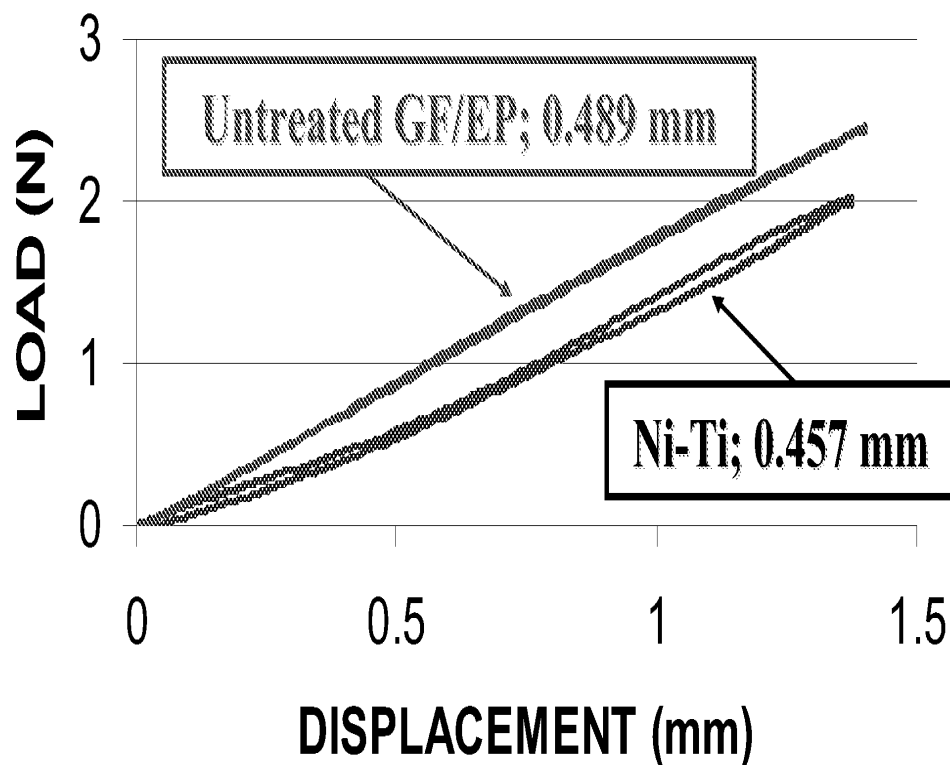
Bending Test

Fixture & Load cell Adaptor



RECOVERY ANALYSIS

Recovery Behaviour (Max. 1.4mm Displacement)

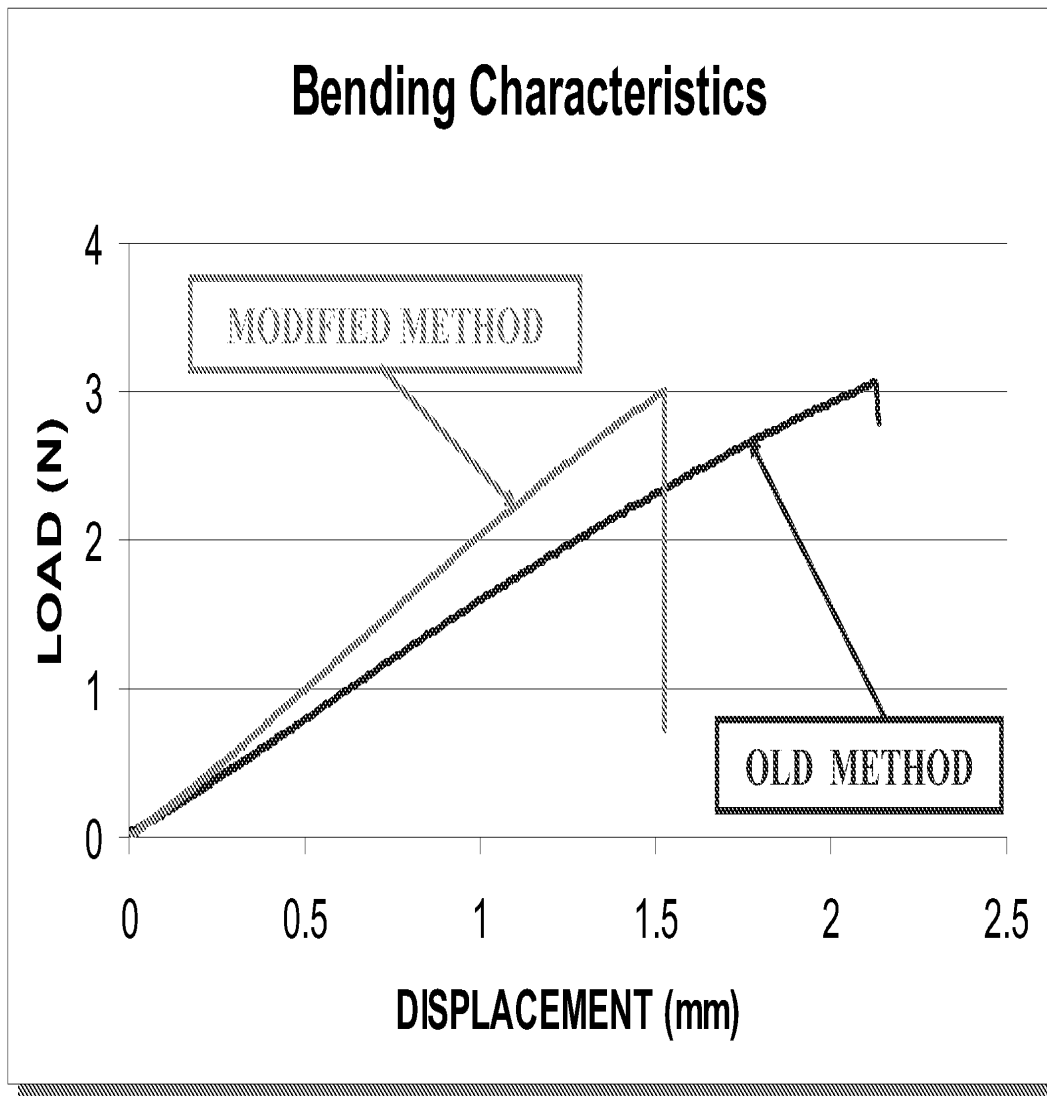


MODIFICATION

- **BEFORE INSERTING GF INTO TUBE....**
 - Apply resin to GF
 - Squeeze impregnated GF
 - Similar to using rollers in pultrusion systems; better impregnation of resin.
 - Brush GF again and then insert into tube

CROSS-SECTION

BENDING CURVE



WORK PLAN.....

- Verify the current modification (GF/EP)
 - If improvement observed, repeat all testing
- Treatment of GF
 - Increase the concentration of coupling agent
 - Determine the optimal concentration
- Increase the GF volume fraction
- Dental resin composite.

